GRScientific

Aquamax KF *Coulometric*

User Manual

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Chapter 1 Introduction

1.1 Instrument Description

Key personnel at G.R. Scientific are recognised experts in Coulometric Karl Fischer titration technology and electrochemistry. They have designed and manufactured Coulometric titrators since the early 1980's and are widely regarded as one of the leading specialists in this technique.

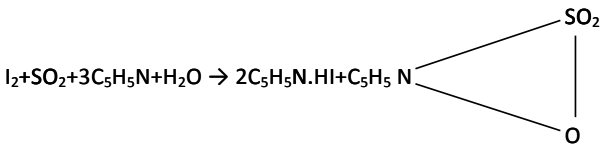
The Aquamax KF *PLUS* is a small foot print titrator with a full numeric keypad for data entry and results manager software for data storage and retrieval. Suitable for use with generator electrodes either with or without frit.

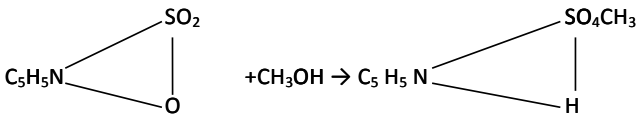
This titrator is a truly versatile model which includes a printer to enable use in areas where connection to a PC isn't available. Low Drift Cell twin port glassware and accessories are supplied as standard. As well as being a stand-alone instrument, this titrator can be combined with accessories such as an Oil Evaporator or a Solids Evaporator system. Suitable for a wide range of applications the Aquamax KF *PLUS* can be used to determine water contents of liquids, gases and solids. An optional carry case for transportation can also be included.

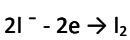
Designed primarily for the oil and power industries, the Aquamax KF *PORTABLE* is the only model on the market with a carry handle attached directly to the unit. It also features a protective cage, built-in printer and single button operation for water standards analysis. Specially designed Low Drift Cell glassware avoids the need for PTFE sleeves or grease on the electrodes to seal titration cell from moisture invasion. A sturdy carry case for transportation in the field is also included.

1.2 Principle of Measurement

Karl Fischer titration is simply a means to measure water content of samples. Modern instruments, such as the Aquamax KF, use the Coulometric principle, whereby the water present in the sample is coulometrically titrated to a predefined end point at which there is a minute excess of free iodine present. Stoichiometrically, 1 mole of water will react with 1 mole of iodine, so that 1 milligram of water is equivalent to 10·71 coulombs of electricity. Combining the Coulometric technique with Karl Fischer titration, Aquamax KF titrators determine the water content of the sample by measuring the amount of electrolysis current necessary to produce the required iodine. This is an absolute technique which does not require calibration of the reagents.





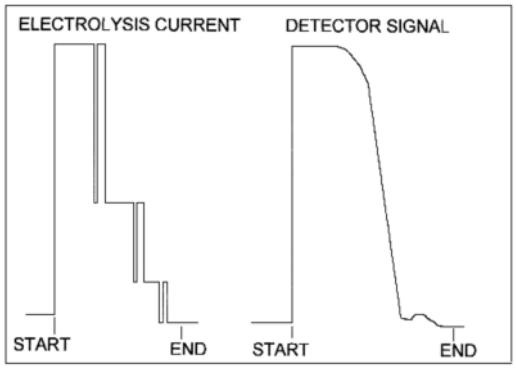


Using the latest pulse current technology and our patented "ACE" control system, (Patent No.GB2370641), the Aquamax KF automatically selects the appropriate titration speed dependent upon the amount of water present in the sample.

The titration speed is reduced as the end point is approached, and when the titration is completed the instrument prints out and displays the results.

For a more detailed explanation of the technique, and a comparison with volumetric Karl Fischer titration, please visit our website, www.grscientific.com

SCHEMATIC OF TYPICAL TITRATION



1.3 Instrument Connections

Various ports are located on the back plate of the instrument. Below is a list of ports that can be found:

(1) Power Supply Port
The power supply port is for connecting to a 18V mains power supply, using the supplied power adaptor.

(2) RS232 Port
For updating titrator software, connecting to other equipment for remote controlling (software specific protocols to be provided).

(3) USB/A Port
For exporting results data to a Removable Flash Drive (memory stick), when running sample tests in the field or remote locations, and subsequent transfer to PC at a later date for loading onto Results Manager files.

(4) USB/B Port
For connecting directly to PC to export data to Results Manager and enable results to be saved on files for future reference. (See Results Manager User Manual section).

Chapter 2 Safety Information

2.1 General Safety Points

GR Scientific products are in conformity with the EU Directives 2004/108/EC and 2006/95/EC. This is based upon the compliance of the products with the harmonised standards: BS EN 61000-4-2:2009; BS EN 61010-2-030:2010; and BS EN 61326-1:2013.

GR Scientific products are manufactured in accordance to RoHS following the EU Directive 2011/65/EU; it does not contain any of the restricted substances in concentrations and applications banned by this directive.

2.2 Electrical Safety

The Aquamax KF Coulometer complies with the international standard IEC 61010.

Only operate this instrument within the mains voltage supply that is specified on the back the instrument, an incorrect mains supply can damage the instrument. Never open the housing of the instrument, only GR Scientific personnel who are trained to service the instrument can open the housing.

There is risk of electrocution if live components are touched.

Always ensure the instrument is disconnected from the mains power supply when connecting or disconnecting cables from the back of the instrument, as electrostatic discharge can build up.

2.3 Solvent and Chemical Safety

When using flammable or irritant solvents and chemicals, follow suitable safety measures to protect yourself and others around. Always use the instrument in a well ventilated area, clean up spilled fluids immediately, and keep flame sources away from the work area. Always follow the safety instructions of the chemical manufacturer and apply the correct countermeasures that are required.

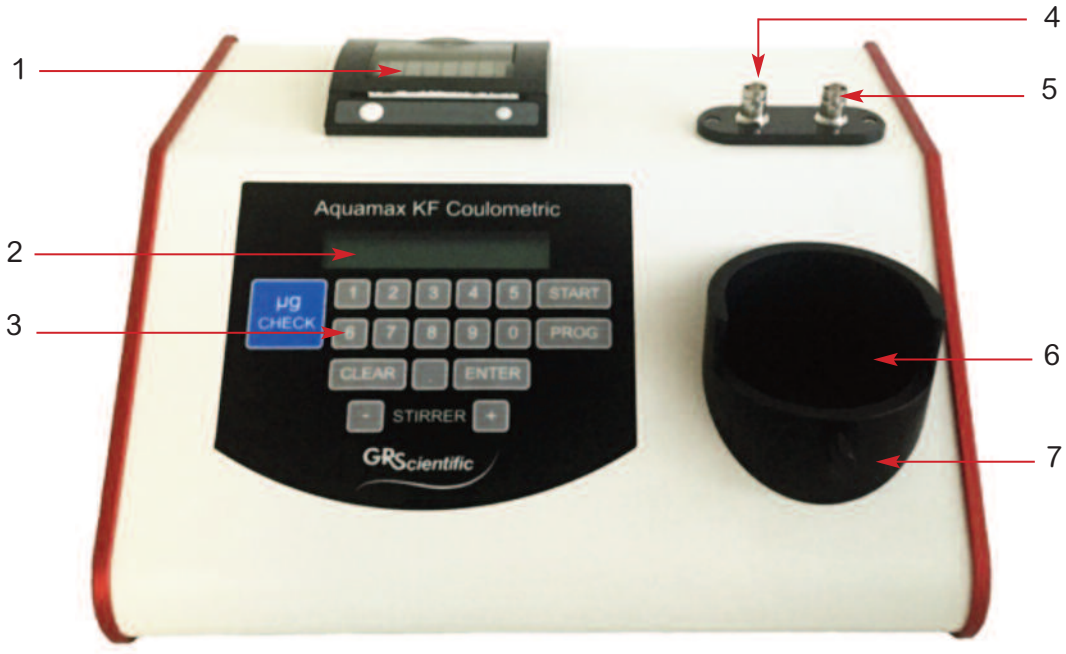
2.4 Recycling and Disposal

This instrument is covered by the WEEE (Waste Electronic and Electrical Equipment) Directive 2012/19/EU to prevent the negative and harmful effects on the environment and public health. For more information on how to dispose of the instrument safely, please contact your local authority, your local waste disposal company or GR Scientific directly.

Chapter 3 Instrument Overview

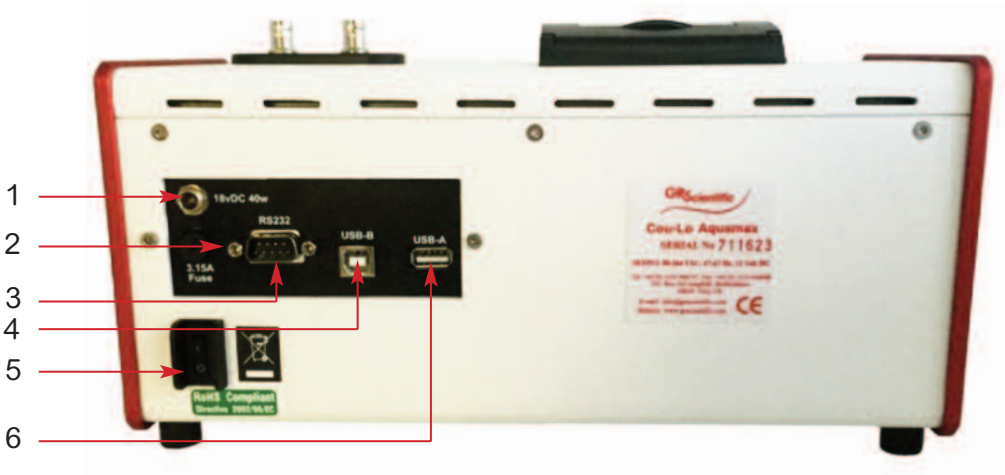
3.1 PLUS Model

3.1.1 Front Panel



(1) Printer
(2) Display
(3) Keypad
(4) Generator BNC Connector
(5) Detector BNC Connector
(6) Vessel Holder
(7) Vessel Retaining Thumb Screw

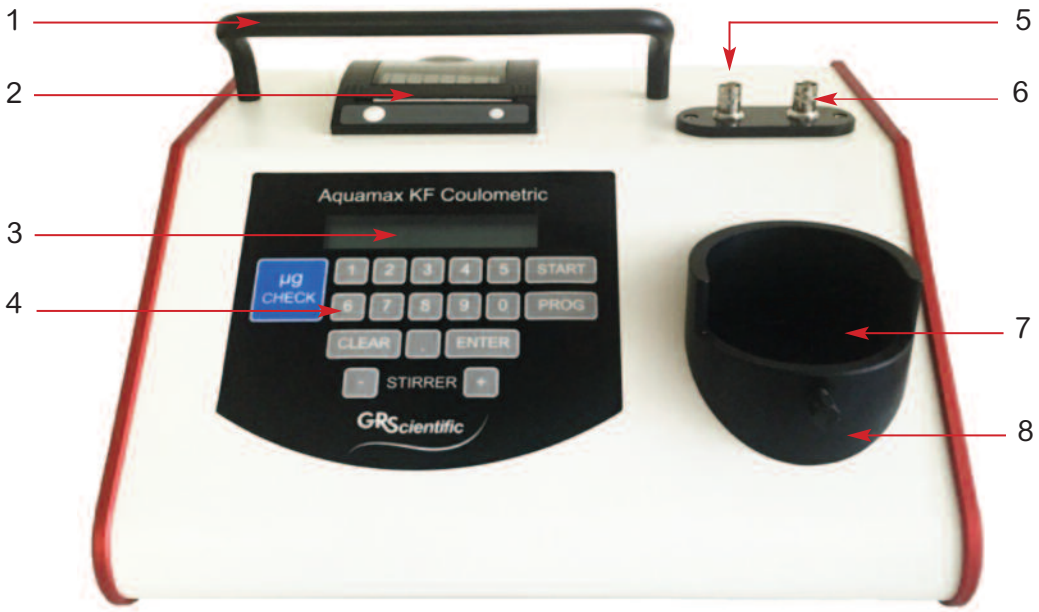
3.1.2 Rear Panel



(1) 18VDC Power Input
(2) Fuse
(3) RS232 Port
(4) USB-B Port
(5) I/O Switch
(6) USB-A Port

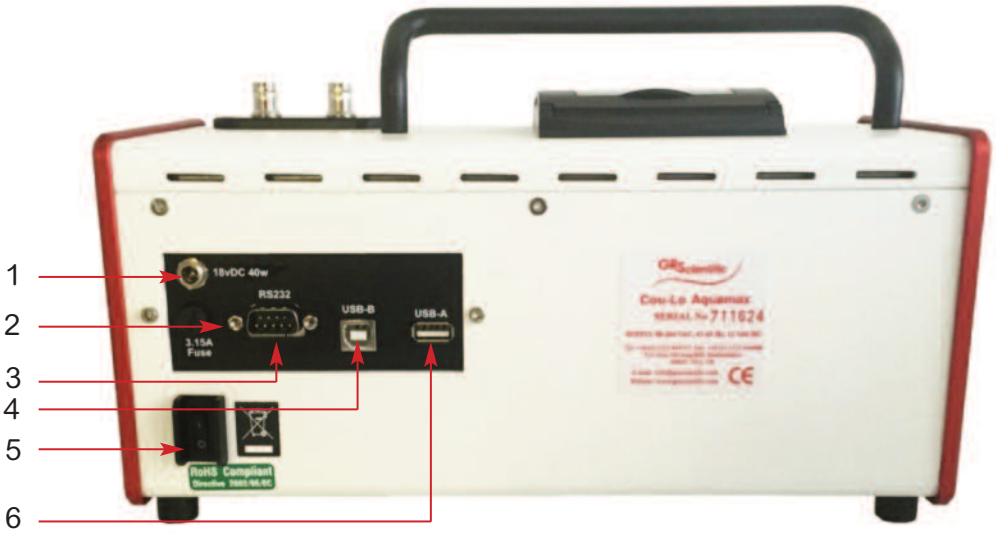
3.2 PORTABLE Model

3.2.2 Front Panel



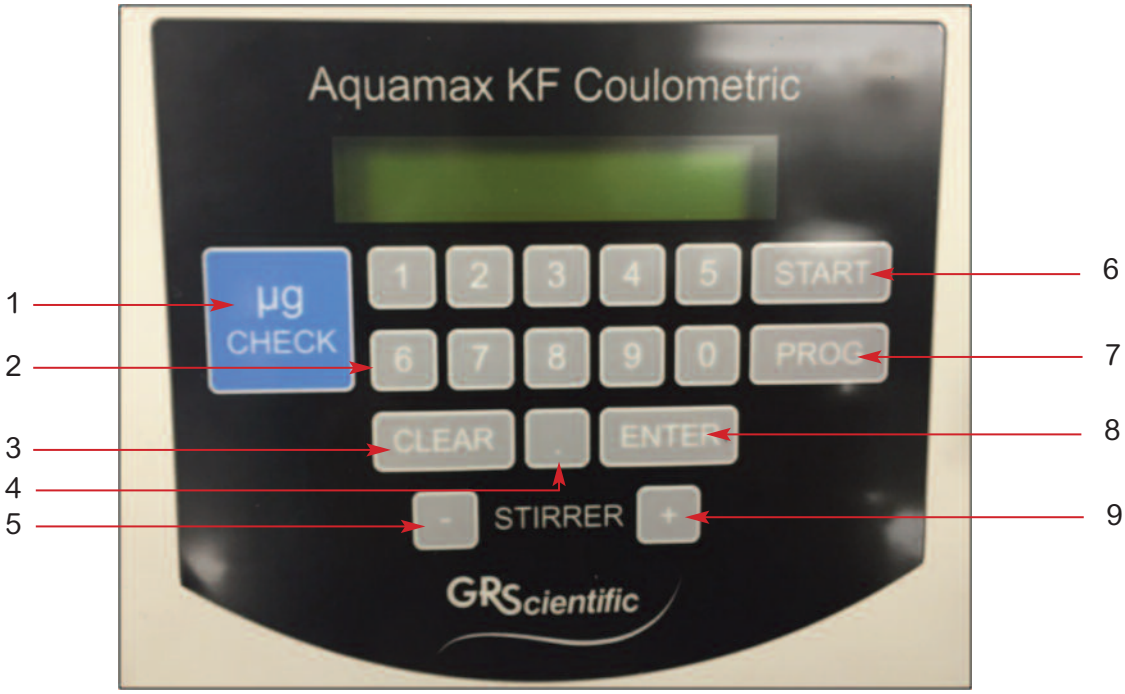
(1) Carry Handle
(2) Printer
(3) Display
(4) Keypad
(5) Generator BNC Connector
(6) Detector BNC Connector
(7) Vessel Holder
(8) Vessel Retaining Thumb Screw

3.2.2 Rear Panel



(1) 18VDC Power Input
(2) Fuse
(3) RS232 Port
(4) USB-B Port
(5) I/O Switch
(6) USB-A Port

3.3 Keypad



(1) µg Check
(2) Alphanumeric 0-9
(3) Clear
(4) Decimal Point
(5) Stirrer –
(6) Start
(7) Programme
(8) Enter
(9) Stirrer +

Chapter 4 Instrument Set Up

4.1 Receiving an Aquamax KF Coulometric

4.1.1 Packaging

The Aquamax KF Coulometric is packaged in suitable protective packaging to ensure that no damage occurs during transit, only this packaging is suitable for the safe transportation during transit. Please keep this packaging as transit of the instrument may need to occur in the future.

4.1.2 Checks

Once you have received the instrument, immediately check to make sure that there is no damage and no parts are missing. A delivery note will be included within the package to use to compare with the shipment.

Ensure that inside the shipment has a calibration certificate for the instrument, if not please request this from the manufacturer.

4.1.3 Location

Locate the instrument within the work area where it is free from excessive:

- vibrations
- direct sunlight
- contamination from chemicals
- explosive environments
- corrosive atmospheres
- major temperature fluctuations

4.2 Connecting the Power Supply

The Aquamax KF Coulometric is powered by an external mains source, with a power pack to ensure this connection is the correct voltage of 18V DC for the instrument.

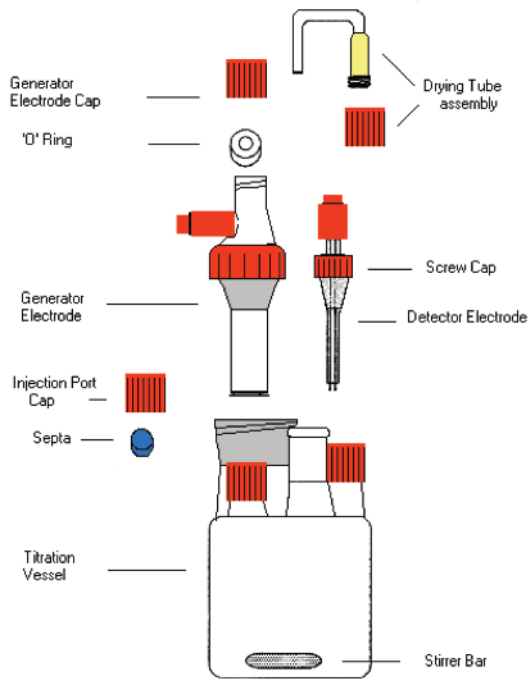
Damage may occur beyond repair and the user may be subjected to electric static shocks if the incorrect power is supplied.

Note: Refer to Chapter 2 for more safety information.

To avoid risk of electrocution, make sure the I/O switch is set to OFF before connecting the power supply.

4.3 Setting Up the Glassware

4.3.1 Glassware Assembly



4.3.2 Connecting to the Aquamax KF Coulometric

Note: This is shown without the protective cage and carry handle, but the same procedure applies.

(1) Position the titration vessel on the titrator.

(2) Connect the generator and detector leads to the electrodes.

Note: The glass joints do not require PTFE sleeves or grease. The special screw fittings seal the vessel from the atmosphere and are easily released, even after several weeks.

(3) Position the electrodes on the titration vessel and tighten by hand.

(4) Connect the generator and detector leads to the BNCs.

(5) Position the drying tube with desiccant and tighten by hand.

(6) Place the stirrer bar inside the titration vessel.

(7) Fit injection septas into the injection port caps and screw tightly by hand onto the injection ports.

The assembled titration cell is now ready to be charged with reagents prior to use. Please be sure to assemble correctly every time the cell is cleaned or re-filled. It is recommended that the titrator is **NOT** switched on until after the titration vessel has been charged with reagents. This will avoid damage being caused to the electrodes by the stirrer bar. Avoid twisting the electrode leads otherwise they may become damaged.

4.3.3 Filling and Changing Reagents

Aquamax KF Coulometric Titrators have been designed to operate with all major Coulometric Karl Fischer reagents. For most routine applications generator electrodes with frit/diaphragm are used in conjunction with 100ml of anode reagent and 5ml of cathode reagent.

When analysing Transformer oils, Crude oils and other petroleum products, we recommend using specially formulated anode reagents which contain other solvents to improve sample miscibility and solubility. There are also single Coulometric reagents available for use with fritless generator electrodes.

Note: When analysing samples of Ketones, amines or others which may interfere with the reaction it is advisable to use specialised reagents which can be obtained from various suppliers.

Although reagents can be poured into the titration vessel whilst it is located on the titrator, we recommend that the vessel, electrodes and electrode leads are removed from the instrument whilst this procedure is performed to avoid reagent spillage onto the instrument casing.

Note: Any spillage onto the instrument casing should be wiped off immediately to avoid damage or staining.

Remove the drying tube and injection septa. Using the funnel supplied, charge the titration vessel with 100ml anode reagent (this is approximately to the lower line on the vessel).

Also using the funnel, charge the inner chamber of the generator electrode with 5ml of cathode reagent. This is not required if using fritless generator electrode.

Note: It is not necessary to clean the funnel between reagents.

Reconnect the drying tube and injection septa so that the titration vessel is sealed from ingress of atmospheric moisture.

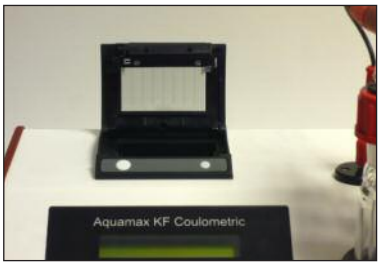
Locate the complete titration vessel onto the titrator and connect the electrode leads onto the appropriate sockets.

There are some bi-products formed in the cathode chamber which can react with the iodine in the anode chamber and this can cause longer preconditioning times. It is advisable to have the cathode reagent 2- 3mm lower than the anode reagent when first charging the titration vessel to speed up preconditioning.

The Aquamax KF titrator is now ready to be switched on.

4.4 Loading the Printer

(1) To load the paper into the printer, first open the paper roll holder lid above the print head and remove the old roll.



(2) Insert the paper roll into the compartment.

Note: To avoid damage to the printer mechanism it is advisable to only use the correct thermal paper rolls, see Chapter 10 Parts and Accessories.

(3) Gently pull the paper roll so it hangs over the printer edge.

(4) Close the printer lid and remove the excess paper.

4.5 Connecting a USB Device

To use the memory store, insert Flash Drive into USB port A, titrator will see it and a symbol will appear on the display.

The symbol will appear blacked out while the drive is being written to and then clear when idle.

The drive should only be removed when in the idle mode.

To read data saved on drive insert it into PC USB port. Start Results Manager, load selected file and then save in the format you require.

Note: See Chapter 8 for full instructions on Results Manager.

Chapter 5 Basics of Operation

5.1 Switching ON and OFF

The Aquamax KF Coulometric has one I/O switch and is powered by either a mains power supply using an adaptor, or a built-in battery for power.

Note: Refer to Chapter 4.2 for power supply connections.

To avoid risk of electrocution, make sure the I/O switch is set to OFF before connecting the power supply.

5.2 Setting the Date and Time

(1) Switch on the Aquamax KF titrator and wait for it to display the following:

(2) Hold down the [START] key until the Aquamax KF titrator displays:

(3) Use the [.] to move the cursor across the screen.

(4) Use the numerical keys to set / alter the required number.

(5) When completed press the [ENTER] to save the changes and exit.

5.3 Preconditioning

(1) Assemble glassware (see Assembly Instructions)

(2) Charge vessel and generator electrode with reagents (see Filling & Changing Reagents)

(3) Locate assembled titration vessel onto the Aquamax KF titrator

(4) Connect electrode leads

(5) Connect mains lead / power cord

(6) Switch ON (using switch on rear panel) and wait for display to show:

Note: If required adjust the stirrer speed using the arrow keys on the front of the instrument.

(7) Press [START] and allow the instrument to precondition (equilibrate).

(8) At the end of the precondition period the display will show:

(9) After the initial drift has been calculated, in micrograms of water per minute, the Aquamax KF is ready for operation.

The background drift value will usually slowly decrease with time as the titration vessel becomes drier and more stable. Although it is possible for the titrator to be used at high drift values, it is advisable to wait until the drift value is below 20µg/minute, and stable, before commencing, especially for low water content samples in the ppm ranges.

The lower and more stable the drift – the more accurate the result.

5.4 Program Functions

When the Aquamax KF titrator displays Ready you can enter titration parameters, recall or edit existing methods, enter new method files, etc., by using [PROGRAM], [ENTER], [CLEAR], and [.].

KEY	FUNCTION
[PROGRAM]	Press [PROGRAM] to scroll through menus listed.
[ENTER]	Allows parameter values for the displayed menu to be entered into memory.
[CLEAR]	1. If you have finished entering data, press [CLEAR] to return to the Ready condition. 2. If you have started to enter an incorrect value, press [CLEAR] to return to the previously stored value.
[.]	Scrolls through individual parameters in each menu. When the required parameter is display, press [ENTER] to store it.

Menu List

Sample ID Number	up to 8 digits
Recall Method	method number 1 – 9
Result Format	µg, ppm, mg/kg or % water
Calculation Mode	V/SG, W/w, W/K or V/v
Printer Mode	off, every result or full report
Statistics Print	up to 99 runs, will print min., max and mean.
Print Drift	on or off
Start Delay Time	up to 30 minutes
Min. Titration Time	up to 30 minutes
End Sensitivity	0 – 9 seconds
Beep Mode	off, key press, end titration or both
Language	select language
Generator Electrode	with frit, without frit
Save Method	store up to 10 methods

Note: See Chapter 5.4 Programming A Method for an explanation of each Menu List parameter.

5.5 Programming a Method

After the initial drift has been calculated, in micrograms (µg) of water per minute, the Aquamax KF titrator is ready for operation. It is now possible to step through the PROGRAM parameters and enter the required calculation data.

Press [PROGRAM] until the required option is displayed then simply follow the Flow Chart in Chapter 5.5 to enter the specific data values. Pressing [PROGRAM] will move you on to the next prompt, pressing [CLEAR] will return to Ready condition. It is not necessary to step through all of the programme functions for every method, for example, if you only require to change the Beep Mode from Off to Both – simple press [PROGRAM] until display shows Beep Mode, press the [.] until display shows "Both", then press [ENTER] and [CLEAR] to return to Ready.

Sample ID Number

Press [PROGRAM] Display will show: Key up to 8 numbers Press [ENTER]

If you do not want to enter any further parameters simply press [CLEAR] to go back to the Ready condition.

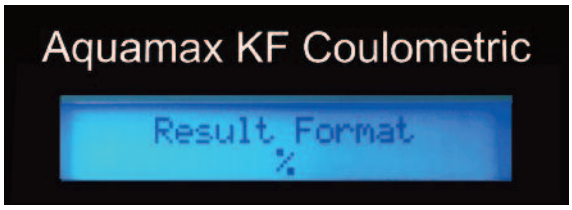
Press [PROGRAM] if you want to continue entering other parameters.

Result Format

Press [PROGRAM] Display will show:

Results can be selected in µg, ppm, mg/kg or % water.

If you want to select the format already stored in memory, and shown on the display, press [PROGRAM] and the instrument will go to the next prompt. If you want to change format to give results in ppm, mg/kg or % you can scroll through these options by pressing [.] until the required format is displayed, and then press [ENTER] to store this in memory and [PROGRAM] to move onto the next prompt. If you have selected a result format in ppm, mg/kg or % the instrument will prompt you for the necessary calculation mode and data values.



The display shows the calculated water content according to the programmed Result Format and Calculation Mode program settings, i.e. whatever has been chosen to be shown/printed at the end of the titration. The only exception to this is in the W/w mode when the Tare Weight has not yet been entered. In this case the display will show the µg count. The [PROGRAM] key can be used to enter the Tare Weight at any time during the titration; once this has been done the display will start to show the calculated result in ppm, mg/kg or % water.

Calculation Mode

Aquamax KF Titrators have four standard calculation modes in memory. Each of these allow the instrument to divide the microgram count by the gram weight of sample introduced so that the calculated result can be printed out in either ppm or percentage of water.

- Press [.] to scroll through the options that are available.
- Select the calculation mode required and press ENTER.
- The display will prompt you with the appropriate parameters.
- Enter the numerical value for each parameter and press ENTER.

The four standard calculations are:

1. Weight difference W/w

Note: This is the most accurate calculation and is suitable for almost all types of samples, powders, liquids, gases, etc.

$$\text{Result (ppm or \%)} = \frac{\text{microgram count}}{W - w}$$

Where: W = Total weight of sampler + sample (gm)

w = Tare weight of empty sampler (gm)

After entering the total weight (W) the instrument will return to the Ready condition. The Tare weight (w) can be entered at any time during the titration by pressing [PROGRAM], or at the end of titration. For ease of operation it is possible to enter the Total weight (W) as 0.0000gm, then place the full sampler on the balance and tare to zero.

Thereafter it is only necessary to enter the weight difference (tare weight, w) for each titration. This procedure reduces potential data entry errors.

2. Weight/Dilution ratio W/K

Note: Use this method if you need to dissolve a sample in another solvent. Suitable for samples which will not dissolve in the anode reagent or which might interfere with the KF reaction).

$$\text{Result (ppm or \%)} = \frac{\text{microgram count - bl}}{\text{W x K}}$$

Where: W = Weight of sample dissolved (gm)

$$\text{K} = \frac{\text{Total volume of diluent + sample}}{\text{Volume injected into Aquamax KF}}$$

bl = micrograms of water for same injected volume of diluent (µg)

e.g. Accurately weigh approximately 2 gm of sample into a 20ml stoppered graduated flask. Make up to the volume line with suitable solvent / diluent. Using appropriate syringe inject 100µl of diluted sample into the titration cell.

Calculation parameters for this example would be:

$$\text{W} = 2\text{ gm}$$

$$\text{K} = \frac{20\text{ml}}{100\mu\text{l}}$$

bl = microgram count of 100µl injected volume diluent

3. Volume / Specific Gravity V/SG

Note: The most widely used technique for liquid samples. However, care must be taken to ensure removal of all air bubbles from the syringe.

$$\text{Result (ppm or \%)} = \frac{\text{microgram count}}{\text{V x SG}}$$

Where: V = volume of sample (ml)

SG = specific gravity

4. Volume V/v

$$\text{Result \% volume} = \frac{\text{microgram count}}{\text{V x 1·0}}$$

Where: V = volume of sample (ml)

This is essentially the same as V / SG mode, except that the sample SG is fixed at 1·0.

Printer Modes

Selecting this program allows the printer to be turned ON or OFF. It also enables you to print out the titration parameters being used at that time. Simply scroll through the options using [.] until the required mode is displayed, then press ENTER.

Options available:

Off	-	No result print out.
Every Result	-	Will print result after every titration, first result also prints all calculation input data.
Full Report	-	Will print results after every titration and then print out statistical data after a selected number of titrations.

Statistics Print

The Aquamax KF titrator can be programmed to print out the statistical data from a series of results when using "Full Report" print mode. If no number has been selected, the Aquamax will automatically clear after 99 runs. If 3 or more runs have been selected the Aquamax will print out the minimum, maximum and mean values.

Print Drift

The Aquamax KF titrator can be programmed to print out the drift value for each titration. This is a useful indicator of titration cell stability. If drift is above 25 µg / minute print will show **XX**. This is a warning that a high drift value was present at start of the titration.

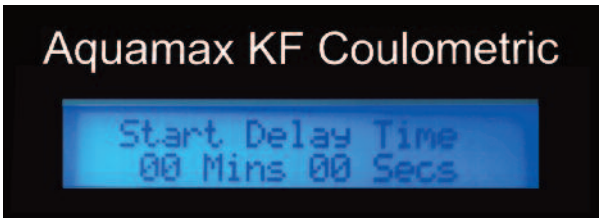
Delay Times

The Aquamax KF titrator has been programmed to accept two different types of delay times, one at the start of titration and one at the end as a minimum titration time.

Start Delay Time

The Aquamax KF titrator has a preset delay of five seconds which allows samples to be dispersed into the reagents before the titration starts. Some samples, particularly powders and viscous samples may need additional time to fully dissolve. By selecting the start delay program, you can increase this time by up to 30 minutes.

Display shows:



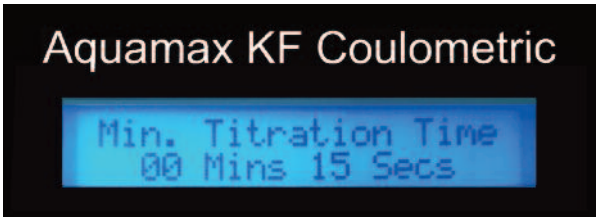
Select required time delay and press [ENTER].

Whilst a delay time is in operation, the display will show "add sample" and the clock counter will show the time remaining before the start of titration. A start delay can be overridden by simply pressing [START].

Minimum Titration Times

For some applications, such as certain gas samples or when using the Aquamax KF titrator combined with an oil evaporator or solids evaporator system, it can prove useful to delay the end point of the titration. By selecting this parameter you can increase the minimum titration time by up to 30 minutes.

Display shows:



Select required minimum time and press [ENTER].

End Sensitivity

The sensitivity of detector signal as the end point is approached can be set from 0 – 9 seconds. The default value is 3 seconds and there are very few occasions when this may need to be altered.

Decreasing this value can prove useful if samples cause interference, e.g. some Lube Oil samples.

Beep Mode

Four options are available for the audible alarm (beeper) and they can be scrolled through using [.] until the required one is displayed. Then press [ENTER].

The options are:

Off	-	Beeper inactive
Key Press	-	Beep once every time a key is pressed
End Titration	-	Beep three times at the end of a titration
Both	-	Beep after every key press and three times at the end of a titration

Language

The Aquamax KF titrator can be programmed to show all display prompts and print outs in various languages. Use [.] to select language then press [ENTER].

Generator Electrode

The Aquamax KF titrator can be operated using generator electrodes with or without frit (diaphragm). Use [.] to select either with or without frit then press [ENTER]. The titration electrolysis current settings are automatically adjusted for each style of generator and appropriate reagents.

Save Method

If you want to save a method for future use, this can be done by pressing a numerical key, 0 - 9, and then [ENTER].

All titration parameters, printer mode, beeper mode, etc. are stored automatically.

Display then shows:

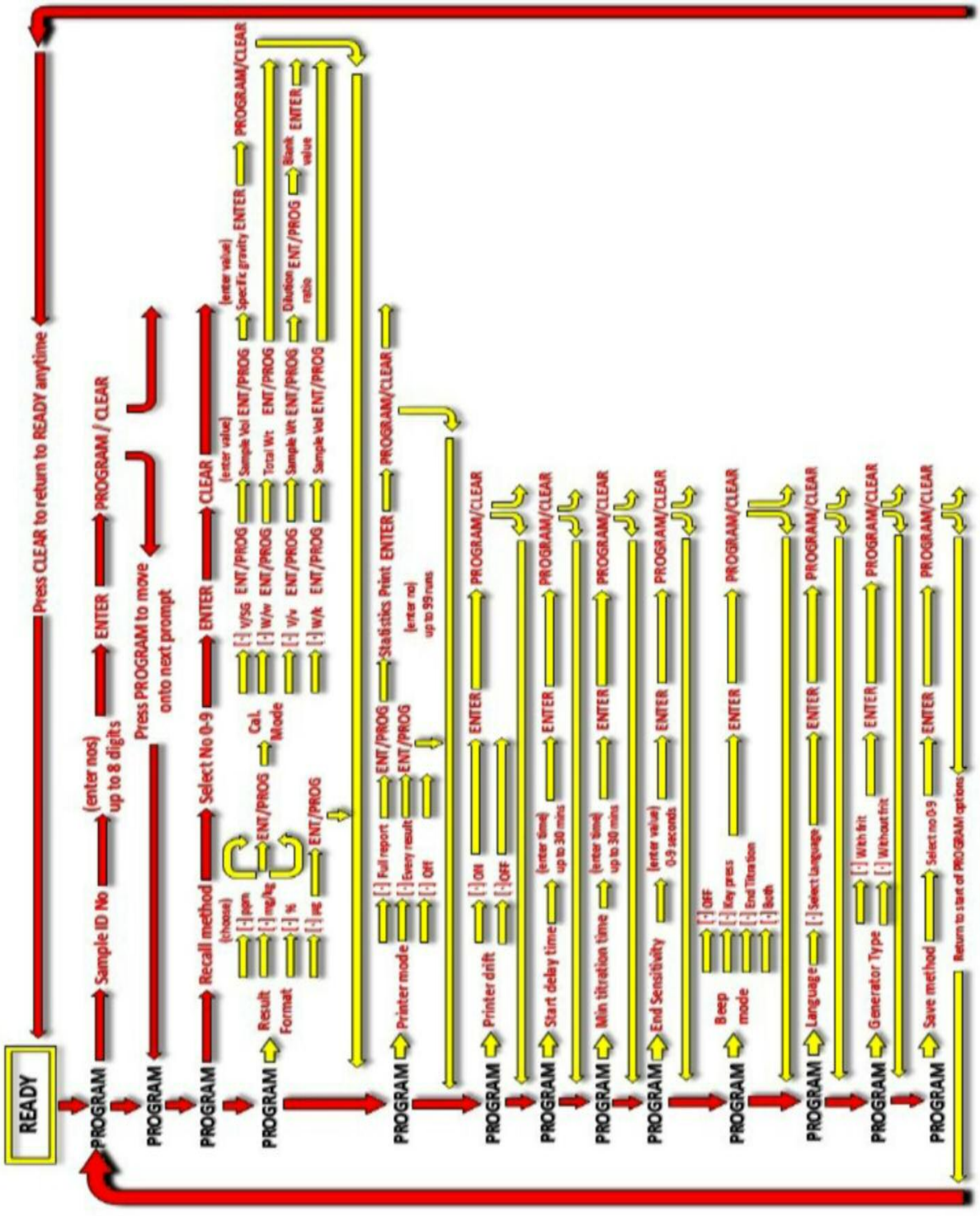
Then press [CLEAR] and the instrument will revert back to the Ready condition.

Recall Method

If any methods have been saved to memory then the first prompt (before Sample ID number) will be “Recall Method.” When this prompt is displayed, simply press the number of the method file you wish to recall and press [ENTER]. There are 10 method files available numbered from 0 to 9. The titration parameters etc will all be automatically recalled. Press [PROGRAM] to view the recalled method parameters or press [CLEAR] and the instrument will go back to the Ready condition.

If you do not want to recall a method, press [PROGRAM] to view the next prompt.

5.6 Program Flow Chart



Chapter 6 Running a Test

6.1 Example Applications

Liquid Samples

Liquid samples are usually introduced into the titration vessel by using a syringe. The greater the amount of water introduced into the titration cell will cause the titration to take longer and also consume the reagents more quickly. It is recommended that the smallest representative amount of sample be used to ensure fastest titration speed and maximum sample throughput before reagents have to be replenished. Optional gas tight syringes and luer needles are available from GR Scientific. With volumes of 0.25, 1.0 and 5.0ml these are suitable for injection of oil samples and water standards. Other volume syringes are commercially available from various suppliers. Please ensure that the needle is long enough to reach into the anode reagent.

This guide may be of assistance when selecting injection volume of samples:

Expected Water Content		Suggested Sample Size	
From	**To**	**From**	**To**
1 ppm	10 ppm	1.0 ml	2.0 ml
10 ppm	100 ppm	1.0 ml	2.0 ml
100 ppm	500 ppm	0·5 ml	1.0 ml
500 ppm	1000 ppm	0·5 ml	1.0 ml
0·001 %	0.01 %	1.0 ml	2.0 ml
0·01 %	0.1 %	1.0 ml	2.0 ml
0·1 %	0.5 %	0·5 ml	1.0 ml
0·5 %	1.0 %	0·5 ml	1.0 ml
1·0 %	5.0 %	0·2 ml	0.2 ml
5·0 %	More Than	Less Than	0.1 ml

Note: These volumes are only a guide. You may wish to experiment with sample size to determine the optimum balance between repeatability, reagent depletion and speed of analysis.

Crude Oils

(IP 386 & 438, ASTM D1533, D4928, D6304, API MPMS Chapter 10.9, ISO 10337 & 12937)

Most Coulometric reagents contain Methanol as the main solvent and as such it is possible that waxy deposits from crude oils can “drop out” and contaminate the electrodes, requiring more frequent and thorough cleaning of the glassware. This problem can be reduced by using Coulometric KF Anode Reagent which has been specially formulated for this application. The most common interference in crude oil samples is usually caused by Mercaptans or Sulphides. Samples containing alkyl groups will react stoichiometrically so that:

- 100 ppm Mercaptans would show as approximately 30 ppm water
- 100 ppm Sulphides would show as approximately 50 ppm water

In normal practice oils containing less than 500 ppm Mercaptans or sulphides are regarded as having no appreciable effect. Repeatability problems are usually caused by sampling inaccuracies or improper homogenisation of the sample.

Transformer/Insulating Oils

(ASTM D 1533, BS EN 60814, IEC 60814)

Due to the viscosity of these samples, and the fact that typical water content levels are in the range 5 – 40 ppm, it is usually necessary to inject sample volumes of 1 ml. This means that up to 50 samples would fill the titration cell. Provided that the other criteria governing reagent life, (total water titrated and reagent age), have not been reached, then it is possible to switch off the titrator and allow the oil to separate from the reagent. The oil can then be siphoned off and the instrument switched on again. After this operation it may take up to 30 minutes before the instrument settles down to a stable baseline. If it takes longer than 30 minutes then it is advisable to clean the cell and recharge with fresh reagents.

Turbine/Lubricating Oils

For most Lube Oil applications a sample size of 1ml is sufficient. However, if it is suspected that the water content is above 1 %, then a sample size of 0·2 to 0·5 ml is suggested.

Some lubricating oils contain additives which can react with the KF reagents interfering with the chemical reaction. For these applications we suggest using the Aquamax KF *PLUS* in conjunction with an oil evaporator such as the Aquamax KF model 510000, as per ASTM D6304.

Ketones and Amines

These can react with the Methanol present in most Coulometric reagents resulting in inaccurate results and, in extreme conditions, titration end point being unattainable. For samples containing Ketones, amines or Aldehydes the use of specially formulated Coulometric reagents for Ketone samples is recommended.

Powder Samples

One of the main advantages of the Coulometric technique is that reagents do not have to be replenished after every analysis; therefore the introduction into the titration cell of solid samples, which do not dissolve, is not recommended.

Three options available:

- Powder samples which readily dissolve in the Anode reagent can be introduced using the optional powder sampler, part no. 503069. Calculation mode W/w.
- Powder samples which cannot be dissolved in the Anode reagent should be extracted or dissolved in a suitable solvent and then an aliquot injected. Calculation mode W/K.
- If neither of above methods are suitable you may need to use a solids evaporator system in conjunction with the Aquamax KF *PLUS* titrator.

Gas Samples

Gas samples can be introduced by fitting a needle onto the gas sampling cylinder and selecting Calculation mode W/w.

Alternatively, gas samples can be bubbled through the titration vessel and the passed volume measured. For this method we suggest using the gas analysis kit (503074) and a wet gas flow meter connected to the outlet of the titration vessel.

(1) Fit the gas inlet and outlet adapters in place of the injection septa.

(2) Remove the drying tube and seal the top of electrode using the injection port septa and screw cap.

(3) Connect outlet adapter to wet gas flow meter.

(4) Flush the sample lines and allow instrument to stabilise.

(5) Press [START] and allow gas sample to flow through the cell at an approximate flow rate of 0·5 litres/minute.

(6) Turn off gas flow and allow titration to reach end point.

Calculation

$$W = \frac{G \times (273 + t) \times 22{\cdot}4}{V \times 273 \times 18}$$

Where: W = Moisture content
G = Microgram count
V = Gas volume, litres
t = Water temperature of wet gas flow meter (°C)

6.2 Water Standard Test

In principle, standardization of a Coulometric Karl Fischer is not necessary since the water titrated is a direct function of the coulombs of electricity consumed. However it is recognised that reagent performance can deteriorate with use over time and it is recommended that the titrator is regularly monitored by introducing a known quantity of water that is representative of the typical range of water concentrations being determined in samples. Several of the ASTM methods for water content of crude oil and petroleum products suggest injecting between 1.0 µl and 10 µl distilled water to check reagent performance.

Each of these methods requires that the result be recorded in micrograms of water, not as a calculated ppm or percentage.

The [µg check] button on every Aquamax KF Coulometric Titrator enables this check to be done without the need to reprogram the calculation mode. Simply press [µg check] and the display will prompt "add standard". Introduce 10µl of distilled water (as required by ASTM methods) by syringe and the display and print out will be in micrograms water. This only applies to a single analysis as the titrator will then revert back to the programmed calculation mode for subsequent runs.

6.3 How to Run a Test

Note: Read Chapter 5.4 Programming a Method then use Chapter 5.5 Programming Flow Chart before running any samples.

Example: Transformer oil sample by V/SG

Program the Aquamax KF titrator with parameters for the analysis. For Transformer Oil samples, these parameters are usually:

Result Format	=	mg/kg or ppm
Calculation Mode	=	V/SG
Sample Volume	=	1.0 ml
Sample SG	=	0.875

Other parameters such as Sample ID number, Printer Mode etc are optional.

Analysis Method

(1) Confirm that Aquamax KF titrator is in "Ready" mode
(2) Flush 1.0ml syringe several times (minimum 6 times) with sample
(3) Fit luer needle and flush through with sample
(4) Draw sample into syringe beyond the 1.0ml marking
(5) Invert syringe so that any air bubbles can be ejected through the needle and adjust syringe plunger to the 1.0 ml mark
(6) Wipe off excess sample from outside of needle using a clean, dry tissue or paper towel
(7) Pierce needle through injection septa of titration vessel (1 – 2 cm)
(8) Press START
(9) Push needle into anode reagent and inject sample
(10) Withdraw needle from titration vessel
(11) Read result, in mg/kg (ppm) water, on display and printout
(12) Repeat steps 2 – 11 if duplicate result required

Example: Powder sample by W/w

Program the Aquamax KF titrator with parameters for the analysis.

1. Analysis Method

(1) Confirm that the Aquamax KF titrator is in Ready mode.

(2) Place a suitable amount of sample in the powder sampler and seal with stopper provided.

(3) Weigh sampler.

(4) Press [PROGRAM] until Calculation Mode is displayed then select W/w.

(5) Enter the sampler weight when prompted by display, then press [ENTER] / [CLEAR]

(6) Remove the injection septa port, press [START], and pour the sample inside the titration vessel ensuring that sample does not stick to the inside of the vessel wall. Replace injection septa port.

(7) Reweigh the sampler.

(8) The net weight of sampler can be entered either during the titration by pressing [PROGRAM] or waiting until the end of titration when display will prompt TARE Weight. Enter the weight then press [CLEAR].

2. Analysis Method

(1) Confirm that Aquamax KF titrator is in the Ready mode.

(2) Place a suitable amount of sample in the powder sampler and seal with stopper provided.

(3) Place sampler on balance and tare to zero.

(4) Press [PROGRAM] until Calculation Mode displayed then select W/w.

(5) Enter the sample weight as 0.0 grams, then press [ENTER] / [CLEAR].

(6) Remove the injection septa port, press [START], and pour the sample inside the titration vessel ensuring that the sample does not stick to the inside of the vessel wall. Replace injection septa port.

(7) Reweigh the sampler.

(8) The weight difference of sampler can be entered either during the titration by pressing [PROGRAM] or waiting until the end of titration when display will prompt TARE Weight. Enter the weight then press [CLEAR].

6.4 Drift Rate Compensation

At the start of any titration, the drift value in the top left corner of the screen (in micrograms per minute), is stored in memory. This ensures that the displayed result is corrected for any ingress of atmospheric moisture during the titration period.

Whilst in the Ready condition, the Aquamax KF titrator continually compensates for any drift caused by atmospheric moisture ingress or reagent decomposition and displays this information, which is updated every 10 seconds. If the drift value is above 30 µg /minute the display will show “Drift Warning START to continue.” This is a warning that the value is high, however the instrument can still be used by simply pressing [START] to continue.

Provided the drift rate is stable then it usually acceptable to continue operation. The Aquamax titration cell design can provide drift values down to less than 5 µg /minute but can also operate at much higher values. The maximum drift value is 60 µg /minute but we recommend operating up to a maximum of 25 whenever possible.

If the drift value is above 60 µg per minute the display will read:

“EXCESS DRIFT - SEE INSTRUCTION MANUAL”

Most common causes of excess drift are:

- Titration vessel not properly sealed (check septa & fittings)
- Reagents almost depleted (clean & recharge the cell)
- Sample introduced before pressing [START] key
- Trace moisture on cell walls

If excess drift occurs, switch the instrument off, remove titration cell from clamp, gently swirl the anode reagent around the cell walls, replace cell in clamp, switch instrument on and allow to precondition.

6.5 Overtitration

If the detector electrode senses too much Iodine in the titration cell, usually caused by instrument being left unoperated for a long period, the display will read :-

“OVERTITRATION - SEE INSTRUCTION MANUAL”

Simply add 3 - 5 microlitres of water, or a small amount of a known wet sample, until the detector signal bars on bottom left of display are activated. Then the instrument will automatically carry out its precondition.

Chapter 7 Results

7.1 Printout Explanation

Aquamax KF Titrators allow you to select different print modes. If you choose to have the printer on, you will receive a hard copy of the result obtained, together with the selected parameters for that particular titration every time a titration is performed.

Options Available:

Off - No result print out

Every Result - Will print result after every titration, first result also prints all calculation input data.

Full Report - Will print results after every titration and then print out statistical data after a selected number of titrations.

The Aquamax KF titrator automatically increments the next run number every time you press the start button. The run counter is reset to zero when power is switched off and back on again. A duplicate set of results can be printed out simply by holding down the start button for 3 seconds.

Results memory is cleared after duplicate print out or when power is switched off.

Print Out Examples

Example of V/SG Calculation

Aquamax KF		
		V xxxx
Serial No.		71000
Calibrated		DD/MM/YYYY
	Run Date	
Run: 1	Run Time	F
Nett Count		0.0 µg
Drift		00
Result Format		%
Calculation Mode		V/SG
Volume		1.0000 ml
Density		1.0000
Res:		0.0000%
Run: 2	Run Time	
Nett Count		0.0 µg
Drift		00
Res:		0.0000%

- Titrator model
- Software version
- Titrator serial number
- Date titrator calibrated
- Date sample run
- Run no. and time of analysis (F = generator with frit)
- Nett microgram count (after drift correction)
- Drift value at start of titration
- Result format selected
- Calculation mode selected
- Sample volume
- Sample density
- Calculated result water content
- Subsequent runs of the same sample and calculation parameters show run number, run time, nett count.
- Drift value and calculated result.

Example of W/w Calculation

Aquamax KF		
		V xxxx
Serial No.		71000
Calibrated		DD/MM/YYYY
	Run Date	
Run: 1	Run Time	N
Nett Count		0.0 µg
Drift		00
Result Format		%
Calculation Mode		W/w
Total Weight		0.0000 gm
Tare Weight		0.0000 gm
Sample Weight		0.0000 gm
Res:		0.0000%

- Run number & time of analysis (N = generator no frit)
- Nett microgram count (after drift correction)
- Drift value at start of titration
- Result format selected
- Calculation mode selected
- Weight of syringe plus sample
- Weight of syringe after sample injected
- Weight of sample analysed
- Calculated result water content

Chapter 8 Results Manager

8.1 Description

Results Manager is a Windows application that allows you to view and print sets of results created by the Coulometric family of Karl Fischer Titrators. It can download results directly from the titrators through a serial port connection, or open result files previously saved to disk.

The Coulometric family of titrators provide a serial interface for the download of test results to our own PC software package, Results Manager, or to third party software running on any equipment with a serial port capable of running at 9600 bps in asynchronous mode. The titrator serial port is configured as a DTE, so connection to a standard PC serial port is achieved using a crossover, or "null modem" cable. The titrator serial port is activated by raising the DCD input pin; this would normally be carried out under control of the software package at the remote end of the link.

Data is transmitted by the titrator in plain ASCII text, with records terminated by a Carriage Return (CR) character. Each record starts with a letter to identify the record type (e.g. Product type and version, Configuration parameter or Result data), a number to identify the exact information contained and one or more items of actual information. Records must be acknowledged by the receiver using a simple positive response record, or they will be retransmitted after a timeout delay; this allows the titrator to move on and send the next record, and causes it to indicate that connection has been established using a special character on its display. The receiving application may also send a negative response to reset the serial interface, causing the entire current set or results to be retransmitted.

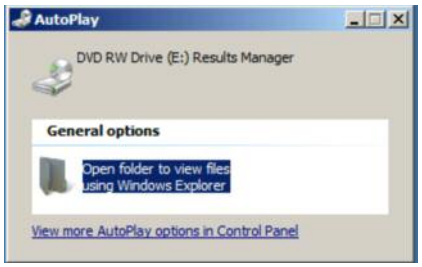
Results Manager Package Comprises of:

Part No. 102026 Results Manager Software CD

Part No. 101009 Interface Cable (connects titrator to computer USB port)

Part No. 101010 512MB USB Flash Drive Memory Stick

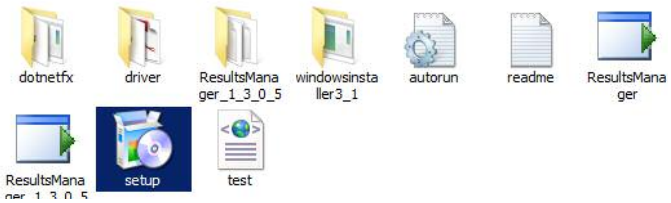
8.2 Installation


AutoPlay
DVD R/W Drive (E:) Results Manager
General options
Open folder to view files using Windows Explorer
View more AutoPlay options in Control Panel

When the Results Manager CD is inserted, it will automatically open an AutoPlay screen in order to install the application.

Note: PCs must have Windows 7 or higher to run the application.

Select setup from the options and follow the process to carry out the installation of the application.



Once this has completed, Results Manager will open automatically. The application can be used as many times with the CD inserted.

To use Results Manager without inserting the CD, copy all of the files from the CD to a folder in My Documents.

8.3 Running Results Manager

With the application running, five buttons across the top of the screen allows the user to carry out the functions that it provides as follows:

Connect

To establish a connection to a titrator linked to the USB port on your PC, click the Connect button.

The first time you do this after running the application, you will be asked to choose the serial port used to make the connection. Desktop PCs will normally have COM1 and perhaps COM2 fitted, while laptops usually have only COM1 to provide functionality on machines without serial ports; these are normally installed in the range COM4 to COM8, although this may usually be changed using the Windows Device Manager. Once connection has been established, the Status display on the screen will change to "Connected" and the text on the Connect button will change to "Disconnect". Clicking it again will disconnect the titrator from the PC.

While connected, Results Manager will download any test results it finds and display them, and will continue to load the results of any further tests that are carried out as long as it remains connected. Because the files that it uses contain single sets of results, when the user starts a new set of measurements on the titrator (i.e. a new sample ID is entered, titration parameters are changed or the end of a fixed run is reached), Results Manager will prompt the user to save the previous set of results before discarding them and loading the new set.

Save

The Save button allows you to save the current set of results to a file on disk. It opens the standard Windows directory and file dialog box to allow you to choose the location and filename for the saved data. Files are stored in XML format (eXtensible Markup Language), which is a standard format widely used to store information in a flexible manner and provide interchange between applications. The extension .xml is used to identify XML files, and will be added automatically to the file name.

Load

The Load button allows you to load a set of results previously saved to disk using the Save button, or transferred from a titrator through some other means. Again, it opens the standard windows directory and file dialog box to allow you to find the directory and file you require, and is configured to display files with the .xml extension. Loading a set of results from file will replace the ones currently held in Results Manager, so if these have not yet been saved you will be asked whether you wish to do so before loading them.

Export

Although many applications can read the XML results files, some cannot. The Export button provides the ability to save the results currently loaded as a text file that can be read by applications such as Microsoft Excel. Again, the standard directory and file dialog allows you to choose the location and name for the file; the results will be stored in CSV format (Comma Separated Values) with the .csv extension.

Print

The Print button allows the current set of results to be printed on any printer connected and supported by Windows. It will open the standard printer dialog, allowing you to choose the printer and set options such as paper type and location. The printout should scale automatically to fit the page, although obviously it has not been possible to test this on every different combination of printer and paper size.

8.4 Display Explanation

Status
Titrator and computer are connected or disconnected.

Model
Manufacturer model and software version.

Serial #
Instrument serial number.

Calibrated
Last date titrator calibrated.

Date
Date analysis recorded.

Results
Total number of results recorded.

Result Format
Selected result format:- µg, ppm, mg/kg or % water.

Calculation Mode
Selected calculation mode:- V/SG, W/w, W/K, V/v.

Results for Sample ID
Samples identification number.

Run #
Sequential count of analysis runs.

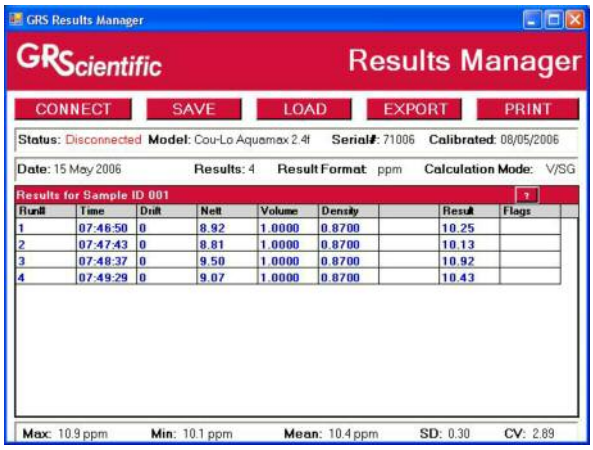
Time
Time analysis recorded.

Drift
Titrator background drift value during analysis.

Nett
The nett microgram count for analysis (after drift deducted).

Prm1, Prm 2 & Prm 3

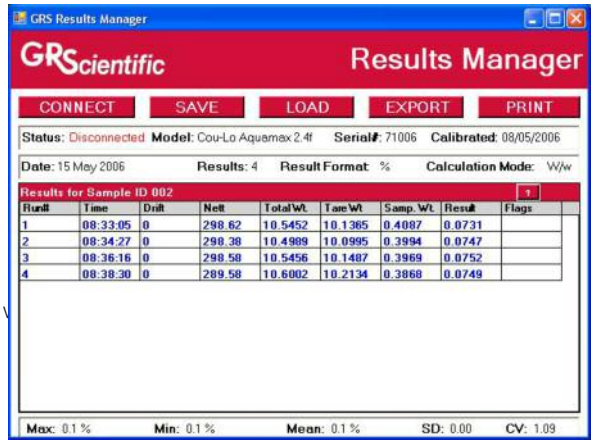
The sample data used for the calculation. These parameters change depending on calculation mode being used. Examples of the three main calculations are shown:


GRS Results Manager
GRScientific
Results Manager
CONNECT
SAVE
LOAD
EXPORT
PRINT
Status: Disconnected Model: Cou-Lo Aquamax 2.4f Serial#: 71006 Calibrated: 08/05/2006
Date: 15 May 2006 Results: 4 Result Format: ppm Calculation Mode: V/SG
Results for Sample ID 001
Run# Time Drift Nett Volume Density Result Flags
1 07:46:50 0 8.92 1.0000 0.8700 10.25
2 07:47:43 0 8.81 1.0000 0.8700 10.13
3 07:48:37 0 9.50 1.0000 0.8700 10.92
4 07:49:29 0 9.07 1.0000 0.8700 10.43
Max: 10.9 ppm Min: 10.1 ppm Mean: 10.4 ppm SD: 0.30 CV: 2.89

Example:

Calculation Mode **V/SG**

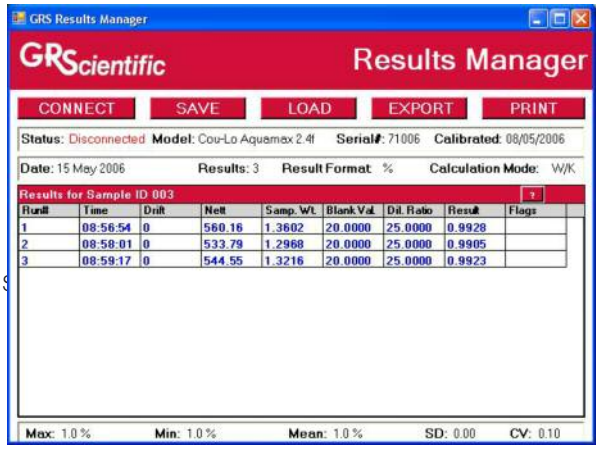
Prm 1 & Prm 2 columns show Volume & Density values


GRS Results Manager
GRScientific
Results Manager
CONNECT
SAVE
LOAD
EXPORT
PRINT
Status: Disconnected Model: Cou-Lo Aquamax 2.4f Serial#: 71006 Calibrated: 08/05/2006
Date: 15 May 2006 Results: 4 Result Format: % Calculation Mode: W/w
Results for Sample ID 002
Run# Time Drift Nett Total Wt. Tare Wt Samp. Wt. Result Flags
1 08:33:05 0 298.62 10.5452 10.1365 0.4087 0.0731
2 08:34:27 0 298.38 10.4989 10.0995 0.3994 0.0747
3 08:36:16 0 298.58 10.5456 10.1487 0.3969 0.0752
4 08:38:30 0 289.58 10.6002 10.2134 0.3868 0.0749
Max: 0.1 % Min: 0.1 % Mean: 0.1 % SD: 0.00 CV: 1.09

Example:

Calculation Mode **W/w**

Prm 1, Prm 2 & Prm 3 columns show Total weight, Tare weight & sample


GRS Results Manager
GRScientific
Results Manager
CONNECT
SAVE
LOAD
EXPORT
PRINT
Status: Disconnected Model: Cou-Lo Aquamax 2.4f Serial#: 71006 Calibrated: 08/05/2006
Date: 15 May 2006 Results: 3 Result Format: % Calculation Mode: W/K
Results for Sample ID 003
Run# Time Drift Nett Samp. Wt. Blank Val. Dil. Ratio Result Flags
1 08:56:54 0 560.16 1.3602 20.0000 25.0000 0.9928
2 08:58:01 0 533.79 1.2968 20.0000 25.0000 0.9905
3 08:59:17 0 544.55 1.3216 20.0000 25.0000 0.9923
Max: 1.0 % Min: 1.0 % Mean: 1.0 % SD: 0.00 CV: 0.10

Example:

Calculation Mode **W/K**

Prm 1, Prm 2 & Prm 3 columns show

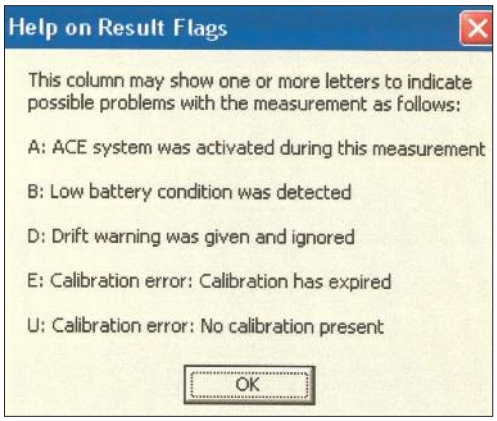
weight, Blank value & Dilution ratio

Result

Calculated result in the selected format.

Flags

If a letter appears in this column, click the question mark (?) for an explanation.


Help on Result Flags
This column may show one or more letters to indicate possible problems with the measurement as follows:
A: ACE system was activated during this measurement
B: Low battery condition was detected
D: Drift warning was given and ignored
E: Calibration error: Calibration has expired
U: Calibration error: No calibration present
OK

8.5 Closing and Uninstalling

Closing Results Manager

The standard close button (the X in the top right corner) will close the application. If results have been downloaded but not yet saved, you will be prompted to save them before closing.

Uninstalling Results Manager

If you no longer require the application, it may be removed from your PC using the Add/Remove Programs utility in the Windows Control Panel. Scroll down the list of applications to the Results

Manager entry and click the button to delete the application.

Chapter 9 Maintenance

9.1 Reagent Life

There are three main factors which govern the life of reagents, and therefore, the frequency of cleaning and recharging the titration cell:

(1) The physical size of the titration cell allows for 50 – 60mL of sample to be added. For the analysis of most liquid samples (e.g. solvents) Aquamax KF titrators normally only requires a few micro litres to be injected, therefore the maximum volume of 50 – 60mL should not become a limiting factor. (Oil samples are an exception and are dealt with elsewhere in this manual).

(2) The total amount of water that can be analysed before saturation of the reagents varies slightly from one reagent manufacturer to another. Typically a 100 ml charge of Anode reagent will analyse up to 1 gm water, whilst a 5 ml charge of Cathode reagent will analyse up to 250 mg water. Considering that the injected sample volume is normally small, and also that the analysis is usually for the determination of low levels of water, these water capacity values are not normally a limiting factor.

(3) Similar to all other Karl Fischer reagents, Coulometric reagents will deteriorate when exposed to sunlight and with increases in temperature. Placing the instrument in direct sunlight or near to a heat duct can decrease the reagent life. As reagents age, the titration speed will reduce and the drift value will increase.

9.2 Cleaning the Glassware

IMPORTANT

The platinum mesh and wires of the electrodes are fragile and can be easily bent or broken. Extreme care MUST be taken when disassembling, cleaning and reassembling the titration cell. The generator electrode is the most expensive part of the Aquamax KF glassware.

DO NOT USE A BRUSH TO CLEAN INSIDE ELECTRODE AS THIS COULD DAMAGE CONNECTIONS.

Coulometric reagents are flammable and toxic, so care should always be taken when handling them and when cleaning the glassware. Whenever possible this should be done in a safe area, e.g. inside a fume cupboard. Reagents should not be recharged while the titration cell is still attached to the titrator.

Under normal circumstances the titration cell can be used for a large number of samples before having to replenish the reagents. The glassware only requires simple cleaning procedure, normally just rinsing with solvent, drying and then re-charging with fresh reagent. Once the reagents have been exhausted, or when the titration cell maximum volume has been reached, it should simply be necessary to:

(1) DISASSEMBLE titration cell (disconnect leads from titrator).

(2) EMPTY the titration cell and generator electrode.

(3) RINSE all parts with methanol. Do not use brush on electrodes.

(4) DRY all parts.

(5) REASSEMBLE glassware.

(6) RECHARGE with fresh reagents.

If the cell is heavily contaminated then it may be necessary to clean it more thoroughly. For oil samples, cleaning with chloroform or xylene is suggested, whilst for salt deposits a water wash may be required. Use whichever solvent is most suited for the sample type. The titration vessel can even be cleaned with hot soapy water and a bottle-brush.

However, after cleaning with suitable solvent, all glassware parts, MUST be rinsed inside and out with methanol.

They can then be dried with a warm air blower, such as a domestic hair dryer, placed in a low temperature oven at 40 – 50 ° C, or left in a desiccator.

After being fully dried, reassemble the titration cell and charge with fresh reagents.

THE MORE THOROUGHLY THAT THE TITRATION CELL IS CLEANED AND DRIED, THE FASTER THE INSTRUMENT WILL STABILISE READY FOR OPERATION AFTER RECHARGING REAGENTS.

Under normal conditions, the Aquamax KF titrator should be ready for operation within 5 – 10 minutes after reassembly, however it could take considerably longer before to completely stabilise if the drying procedure has not been properly carried out.

Generator Electrode – Acid Wash Procedure

If the frit of the generator electrode has become blocked or severely stained with samples such as crude oils, then it may occasionally be necessary to use an acid wash procedure.

(1) Rinse the empty generator electrode with Xylene or other suitable solvent.

(2) Place the electrode in a 250ml conical flask, ensure the electrode is suspended and not resting on the platinum mesh. Add 10ml Xylene to the inner cathode chamber and leave to stand for 10 minutes. Remove the Xylene.

(3) Repeat using 10ml of Methanol then gently swirl to remove residual solvent and remove.

(4) Add 10ml deionised water to inner cathode chamber and allow to stand for 30 minutes.

(5) Remove any remaining water, add 10ml 50/50 HCL and allow to stand for 1 hour. Top up any loss of HCL through the frit. After 1 hour carefully pour the acid into a flowing water stream in the sink, ensuring that the breather hole midway down the side of the electrode is positioned at the top when pouring acid out.

(6) Thoroughly rinse the electrode using a wash bottle containing deionised water. Add 10ml deionised water to the inner chamber and allow to stand for 30 minutes.

(7) Remove any remaining water, add 10ml Methanol and allow to stand for 10 minutes. Ensure that the outer surfaces of the electrode do not come in contact with waste solution gathering in the bottom of the vessel.

(8) Drain remaining solvent from inner chamber and dry electrode.

Note: Wear safety goggles and protective gloves when handling acid.

Chapter 10 Technical Specifications

10.1 PLUS Model

Titration Method:	Coulometric Karl Fischer Titration
Electrolysis Control:	Patented "ACE" Control System GB2370641
End Point Detection:	AC Polarisation
End Point Indication:	Visual Display/Print Out/Acoustic Beep
Titration Vessel:	Low Drift Cell Design, no grease or PTFE sleeves required
Measuring Range:	Possible 1µg – 200mg Water Typical 1µg – 10mg Water
Moisture Range:	1ppm – 100% Water
Max. Sensitivity:	0.1µg
Max. Titration Speed:	2.24mg per minute
Max. Current:	400ma
Drift Compensation:	Automatically Controlled
Precision:	10-100µg ± 3µg, 100µg -1mg ± 3µg, above 1mg ± 0.3%
Start Delay Time:	0-30 minutes, user selectable
End Delay Time:	0-30 minutes, user selectable
Calculation Modes:	Weight/weight (W/w) Weight/Dilution Ratio (W/K) Volume/Density (V/SG) Volume/volume (V/v) (User Programmable)
Display Format:	µg, mg/kg, ppm, %
Print Format:	µg, mg/kg, ppm, %
Statistics:	Max, Mean, Min values up to 99 runs
Method Storage:	10 User Programmable Methods
Sample ID Number:	User Programmable
Stirrer Speed:	Microprocessor Controlled – User Selectable
Languages:	Multi Languages – User Selectable
Calendar/Clock:	Analysis Time & Date Print Out
Data Outputs:	USB & RS232 Ports
Removable Data Storage:	Flash Drive (memory stick)
Data Entry:	18 Key Touchpad
Display:	40 Character Alphanumeric Backlit LCD
Printer:	42 Character High Speed Thermal Printer
Power Supply:	90-264V AC, 47-63Hz
Unit Dimensions:	290 x 255 x 130 mm
Weight:	3kg

10.2 PORTABLE Model

Titration Method:	Coulometric Karl Fischer Titration
Electrolysis Control:	Patented “ACE” Control System GB2370641
End Point Detection:	AC Polarisation
End Point Indication:	Visual Display/Print Out/Acoustic Beep
Titration Vessel:	Low Drift Cell Design, no grease or PTFE sleeves required
Measuring Range:	Possible 1µg – 200mg Water Typical 1µg – 10mg Water
Moisture Range:	1ppm – 100% Water
Max. Sensitivity:	0.1µg
Max. Titration Speed:	2.24mg per minute
Max. Current:	400ma
Drift Compensation:	Automatically Controlled
Precision:	10-100µg ± 3µg, 100µg -1mg ± 3µg, above 1mg ± 0.3%
Start Delay Time:	0-30 minutes, user selectable
End Delay Time:	0-30 minutes, user selectable
Calculation Modes:	Weight/weight (W/w) Weight/Dilution Ratio (W/K) Volume/Density (V/SG) Volume/volume (V/v) (User Programmable)
Display Format:	µg, mg/kg, ppm, %
Print Format:	µg, mg/kg, ppm, %
Statistics:	Max, Mean, Min values up to 99 runs
Method Storage:	10 User Programmable Methods
Sample ID Number:	User Programmable
Stirrer Speed:	Microprocessor Controlled – User Selectable
Languages:	Multi Languages – User Selectable
Calendar/Clock:	Analysis Time & Date Print Out
Battery Low Indicator:	Display & Print Out Indication
Data Outputs:	USB & RS232 Ports
Removable Data Storage:	Flash Drive (memory stick)
Data Entry:	18 Key Touchpad
Display:	40 Character Alphanumeric Backlit LCD
Printer:	42 Character High Speed Thermal Printer
Protective Sleeve	Included
Carrying Handle	Included
Power Supply:	90-264V AC, 47-63Hz 12V DC Car Adaptor/Internal Battery
Unit Dimensions:	290 x 255 x 130mm (Not including carrying handle or protection cage)
Weight:	5kg

Chapter 11 Parts List

11.1 PLUS Model

Part No: 101001

All the following parts are included as standard with the PLUS model, but can be purchased as single items.

101005 – Results Manager Software Package

Comprises of:

- **102026** - Results Manager Software CD
- **101009** – Interface Cable
- **101010** – 512MB USB Flash Drive Memory Stick

401511 – Power Adaptor

503052 – Glassware Set LDC Twin Port

Comprises of:

- **503053** – Titration Vessel LDC
- **503054** – Detector Electrode LDC
- **503055/503056** – Generator Electrode ± frit LDC
- **503057** – Electrode Lead x2
- **503058** – Desiccant Tube LDC & Cap
- **503059** – Pack Injection Septa (10 pack)
- **503070** – Molecular Sieve
- **503071** – Stirrer Bar
- **503072** – Polypropylene Funnel
- **503160** – Paper Roll (Thermal) Single x3
- **503164** – Luer Needle 19 Gauge (Screw Fit)

503063 – Gas Tight 1.0ml Syringe

All the following parts are not included as standard with the PLUS model, but can be purchased as optional extras.

201004 – Carry Case

503062 – Gas Tight 0.25ml Syringe

11.2 PORTABLE Model

Part No: **101016**

All the following parts are included as standard with the PORTABLE model, but can be purchased as single items.

101005 – Results Manager Software Package

Comprises of:

- **102026** - Results Manager Software CD
- **101009** – Interface Cable
- **101010** – 512MB USB Flash Drive Memory Stick

201004 – Carry Case

301203 – Vessel Sleeve

401424 – In-Car Adaptor

401511 – Power Adaptor

503046 – Glassware Set Single Port

Comprises of:

- **503047** – Titration Vessel
- **503048** – Detector Electrode
- **503049/503050** – Generator Electrode ± frit
- **503051** – PTFE Sleeves (10 pack)
- **503057** – Electrode Lead x2
- **503058** – Desiccant Tube LDC & Cap
- **503059** – Pack Injection Septa (10 pack)
- **503070** – Molecular Sieve
- **503071** – Stirrer Bar
- **503072** – Polypropylene Funnel
- **503160** – Paper Roll (Thermal) Single x3
- **503164** – Luer Needle 19 Gauge (Screw Fit)

503062 – Gas Tight 0.25ml Syringe

503063 – Gas Tight 1.0ml Syringe

Chapter 12 Troubleshooting

Overtitration

Detector senses too much Iodine. Usually caused by old reagents or stirrer switched off. Check stirrer speed.

Add 3-5 micro litres of water until detector bars activate, and allow to precondition.

Excess Drift

Indicates excess moisture ingress or condensation on cell walls. Switch OFF. Remove titration cell from clamp and gently swirl anode reagent around the cell walls. Reconnect, switch ON, and allow to Precondition.

Long Preconditioning Time

Normally caused by glassware not being sufficiently dried before assembly. Re-clean glassware and electrodes, dry thoroughly and charge with fresh reagents.

Progressively Lower Results for Same Sample

Moisture contamination of syringe. Flush syringe and needle with sample several times before beginning new test.

Poor Repeatability for Same Sample

Sample size probably too small or moisture contamination of syringe. Increase sample size and flush syringe and needle several times before beginning new test.

Warning Letters on Print Out

Warning letters will be printed after time of each analysis to signify the following:

U – Uncalibrated

E – Calibration expired

B – Battery level low

A – ACE system active

Results Manager

The serial port parameters are set automatically by Results Manager, so a failure to connect to the titrator is most likely to be caused by selecting the wrong serial port when clicking the Connect button. Other possible causes of a connection failure are as follows:

Incorrect titrator software. The serial port functionality is fully implemented in version 2.6a and above of titrator firmware and previous versions may not work reliably or establish a connection at all. Contact GR Scientific or your local agent in order to obtain an upgrade to your titrator firmware.

Note: The firmware version is displayed on the titrator immediately after the unit is switched on.

Chapter 13 Conformity and Warranty

13.1 Declaration of Conformity

GR Scientific products are in conformity with the EU Directives:

- 2004/108/EC Electromagnetic Compatibility Directive
- 2006/95/EC Low Voltage Directive

This is based upon the compliance of the products with the harmonised standards:

- BS EN 61000-4-2:2009 Electromagnetic Compatibility. Generic immunity standard. Residential, commercial and light industry;
- BS EN 61010-2-030:2010 Safety requirements for electrical equipment for measurement, control and laboratory use. Particular requirements for testing and measuring circuits;
- BS EN 61326-1:2013 Electrical equipment for measurement, control and laboratory use. EMC requirements. General requirements.

13.2 Quality Control

The Aquamax KF Coulometric; is adequately and professionally Quality Controlled to ensure that it is fully functional and to ensure that it meets all of the requirements that the instrument supports.

13.3 Customer Support and Service

We want you to maximise the return on your equipment through a service program tailored to your requirements from when you purchase our equipment which includes:

- Support and Repair
- Maintenance and Calibration
- Training and Installation
- Quality Assurance and Certification

Our dedicated service staff are on hand to help in the unlikely event that your equipment develops a fault. Please contact us using the details provided to discuss the next step.

13.4 Warranty

Under the conditions of our warranty, all faults which are proved to be due to material, construction or manufacturing defects, which occur within 24 months of the delivery date, will be repaired or replaced, (at our discretion), free of charge at our premises. Freight costs will be chargeable along with any inspection work which was not necessitated by manufacturing or material defects. Breakages of glassware or electrodes are not covered by this guarantee. In the event of instrument failure during the warranty period, written authorisation must be obtained from the manufacturer prior to shipping.

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